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(71) Applicant: WESTINGHOUSE ELECTRIC CORPORATION
Westinghouse Building Gateway Center
Pittsburgh Pennsylvania 15222(US)

(72) Inventor: Radford, Kenneth Charles
1637 Georgetown Place
Pittsburgh Pennsylvania(US)

(72) Inventor: Carlson, William George
3339 Woodland Drive
Murrysville Pennsylvania(US)

(74) Representative: van Berlyn, Ronald Gilbert
23, Centre Heights
London, NW3 6JG(GB)

(54) **Method of making annular pellets for use as burnable neutron absorbers.**

(57) Annular pellets of burnable poison specifically boron carbide, B_4C, in a matrix of a refractory material, specifically aluminum oxide, Al_2O_3, are produced. The pellets are of small wall thickness. Powders of the Al_2O_3 and the B_4C are milled in a ball mill in water in which a wetting agent, a surfactant and a deflocculant are included to produce a slurry. Organic binders and plasticizers are added. Then the slurry is spray dried in a centrifugal separator. The resulting powder is poured into a mold and a tubular green body is formed by isostatic pressure. The tube may be sintered to size as a whole and then cut into lengths; i.e., pellets, or the green body may be cut into green-body pellets which are then sintered.

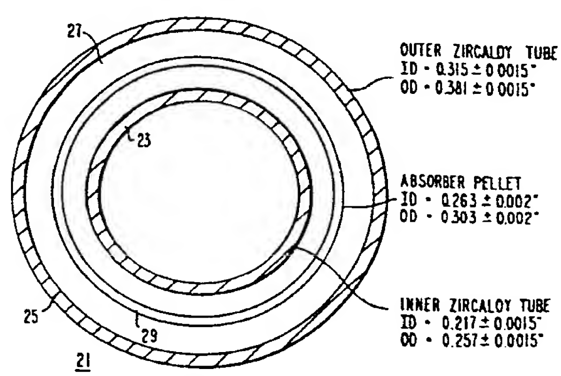

FIG. I

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METHOD OF MAKING ANNULAR PELLETS FOR USE AS BURNABLE NEUTRON ABSORBERS

This invention relates to a method of making annular pellets for use as burnable neutron absorbers in nuclear reactors. This invention relates particularly to neutron-absorbers such as are disclosed in Orr application. United States Patent Application Serial No. 915,691 (Orr et al.), filed June 15, 1978, discloses neutron absorbers including annular neutron-absorber pellets in a closed annular chamber or cavity between coaxial cylinders of ZIRCALOY alloy. The pellets are composed of ceramics including a matrix of a highly refractory material in which is embedded or encapsulated a burnable poison. Typical matrix material are aluminum oxide, Al_2O_3, and zirconium oxide, ZrO_2. Certain isotopes of the elements boron, gadolinium, samarium, cadmium, europium, hafnium, dysprosium, and indium are burnable neutron absorbers. One or more of these elements in their natural state or enriched in the neutron-absorbing isotopes are encapsulated in the matrix. Usually the element, natural or enriched, is encapsulated as a compound. Of particular interest is boron whose isotope boron 10 is a neutron absorber. Typical ceramics are a matrix of Al_2O_3 encapsulating B_4C or a matrix ZrO_2 encapsulating zirconium boride, ZrB_2. B_4C with depleted boron also may serve as a material for a burnable poison. The Al_2O_3 + B_4C ceramic and the ZrO_2 and ZrB_2 ceramic may include natural boron or boron

enriched or depleted in B^{10} with the quantity B^{10} varied depending on the radial wall thickness and density of the pellets and the purpose which they are to serve. In the B_4C with the depleted B^{10}, the B^{10} is set to yield the required B^{10} loading per foot of the pellet. The primary neutron absorber material includes a matrix of Al_2O_3 encapsulating B_4C.

It has been proposed that the annular pellets be produced by forming a green body of powders of the matrix and neutron absorber material, sintering the green body and cutting and machining or grinding the resulting body to size. Typically the finished pellet so produced is about 2 inches in length. In the interest of economy, particularly to avoid excessive scrap in the finishing of individual pellets, and in the interest of practicability, a green body is produced which after sintering may be severed into blanks for forming several green bodies. Typically the cylinder is a tube 7 or 8 inches in length. About 2 to 3 finished pellets are derived from a 7 or 8 inch tube. The practice has been to grind the inside and outside of the sintered pellets. This is a costly and time-consuming operation. It is desirable that the grinding step be dispensed with.

Structurally the annular pellets are subject to critical demands. Annular pellets of very small radial dimension; i.e. thickness, and of very light tolerances are required. The thickness (radially) is typically between 0.020 and 0.040 inches. The typical spacing between the outer diameter of the inner cylinder of ZIRCALOY alloy and the inner diameter of the outer cylinder is relatively small. It is then necessary that the dimensions of the pellet, particularly its radial thickness, shall be maintained within tight limits. Because precise B^{10} loading is essential to reactor operation, the density and wall thickness of the pellets are critical. In the operation of a reactor the nuclear reaction between the

neutrons which bombard the B^{10} results in the formation of helium. In addition the bombardment of the pellets by neutrons displaces the atoms of the ceramic causing it to swell. The ceramics of which the pellets are formed are then porous, typically between about 60 and 80 per cent of theoretical density. The requirement that the density be microscopically uniform applies to the per cent theoretical density. The swelling and the emission of helium subjects the pellets to substantial pressure. It is then required that the pellets have substantial strength so that they can withstand the pressure. It is essential that this requirement be met since the pellets have small radial thickness. Not only must these properties of the pellets be uniform but they must also be reproducible from pellet to pellet of any batch of material for producing the pellets, and from batch to batch. These demands are applicable to the relatively long cylinder from which the pellets are formed. It is necessary that a long cylinder having a truly linear axis about which it is symmetric be formed and that this cylinder have uniform density and wall thickness throughout. Such demands call for sophisticated manufacturing practice.

It is an object of this invention to provide a method for producing economically, burnable neutron-absorber pellets meeting the above demands. It is contemplated that this object will be met by forming a green body from the powders of the components and sintering the green body to size, thus dispensing, to the extent practicable, with a final grinding.

Accordingly, the present invention resides in a method of making an annular pellet for use as a burnable neutron absorber in a nuclear reactor, characterized by mixing a first powder to form a matrix, said powder being selected from one or more of the class consisting of aluminum oxide (Al_2O_3) and zirconium oxide (ZrO_2) with a second powder of a neutron absorbing one or more of the

class of elements consisting of boron, gadolinium, samarium, cadmium, europium, hafnium, dysprosium and indium or their compounds; milling said first powder and said second powder in a liquid to produce a slurry; drying said slurry to produce a dry mass of said mixed first and second powders; adding to said mass of powders means capable of adding strength to a green body to be formed of said mass of powders; depositing said mass of powders into a mold in which said mass may be subjected to isostatic pressure; subjecting said mass of powders in said mold to isostatic pressure to form a green body having the form of said pellet; and heating said green body at a temperature such as to drive off said strength-adding means and to sinter said green body to form said pellet.

In accordance with this invention there is provided ceramic processing including sintering schedules in whose practice annular pellets having the properties and precision demanded are sintered to size. In the practice of this invention a homogeneous ceramic powder of the constituents of the matrix and of the neutron absorber is prepared. During the sintering atoms of the matrix diffuse throughout the original green body and the body shrinks. It is desirable that the ceramic powder be so homogeneous as to preclude the occurrence of inhomogeneous shrinking during sintering. Typically the powder includes Al_2O_3 and B_4C and the content of B_4C in the powder in weight per cent is from 1 to 50.

In the practice of this invention, the Al_2O_3 and B_4C appropriately sized are milled in a ball mill with liquid to produce a slurry. The slurry is spray dried to produce small spheres of the mixed powder. This powder, containing adequate organic binder and plasticizer, is formed into a green body having the shape of the tube. The green body is sintered to produce a ceramic tube from which the pellets are cut. The tube is sintered to size so that the pellets have the required dimensions.

It is an important feature of this invention that the powder is formed into the green body by applying isostatic pressure to the powder. It has been found that by the use of the isostatic pressure precisely dimensioned pellets of the requisite strength and of uniform density and wall thickness, meeting all of the above demands, are produced. The inner diameter of the pellets is precisely dimensioned. The outer diameter usually requires minimal grinding.

In arriving at this invention other modes than isostatic pressure for forming the green bodies were considered. Uniaxial cold pressing results in density variations along the length of the compacted body. The uniform density and wall thickness along the length of the pellet which is demanded could at best be achieved only for a very short pellet, less than ¼ inch in length. This method is not practicable for forming the green bodies. Extrusion of the ceramic tubes would require a large quantity of organic binder and plasticizer material. The sintered body would then have excessive porosity (and low strength) and would have density variations along its length. During sintering uneven shrinkages would also occur so that the cross section of the ceramic tubes would not be substantially perfectly circular and the tubes would be bent. Isostatic pressing is readily controllable. A minimum of organic binder and plasticizer is required to give the green body strength. By appropriate control of the ceramic powder processing, uniform shrinkage during the sintering following the isostatic pressing may be assured. The green tube may then be sintered directly to size. Also, tube length and other dimensions can be made to suit the requirements for rod loading. Typically the length of a pellet is between 1 and 2 inches.

In order that the invention can be more clearly understood, a preferred embodiment thereof will now be described, by way of example, with reference to the accompanying drawings in which:

Fig. 1 is a view, in transverse section, of a burnable neutron-absorber rod with burnable pellets;

Fig. 2 is a flow chart of a method of making the burnable pellets of Fig. 1;

Fig. 3 is a view, in perspective, of an apparatus for drying slurry;

Fig. 4 is a view, in perspective, of a multiple mold for producing ceramic tubes;

Fig. 5 is a longitudinal view, in section, taken along line V-V of Fig. 4;

Fig. 6 is a plan view showing a funnel used for pouring a mixed powder into a mold and its relationship to a mandrel of the mold;

Fig. 7 is a view, in transverse section, taken along line VII-VII of Fig. 6;

Fig. 8 is a fragmental view, in longitudinal section, showing the manner in which the powder is deposited in the mold;

Fig. 9 is a fragmental view, in section, showing the manner in which a plug or cap is inserted in a cavity;

Fig. 10 is a graph showing the typical relationship between the per cent of theoretical density and the pressure for green ceramics;

Fig. 11 is a graph showing the relationship between the sintered per cent theoretical density at two sintering temperatures of green ceramics and the content of B_4C;

Fig. 12 is a graph showing the relationship between the per cent theoretical density of green ceramics and the sintering temperature, for sintering for three different durations; and

Fig. 13 is a graph showing the relationship between the per cent theoretical density of green ceramics and the sintering temperature for different Al_2O_3 powders.

Referring to Fig. 1 a neutron-absorber rod 21 includes inner hollow cylinder 23 and outer hollow cylinder 25, these cylinders being sealed at their ends and defining

an annular chamber 27 in which pellets 29 are stacked coaxially with the cylinders. The afore-mentioned Orr et al. application shows the rod 21 in more detail. Fig. 1 shows typical dimensions of the cylinders 23 and 25 and the pellet 29. It is emphasized that the diameters of the cylinders must be maintained to within plus or minus 0.0015" and the diameter of the pellet to within plus or minus 0.002". The concern is to produce these pellets to size with a minimum of grinding or other machining processes.

The pellets 29 are ceramics produced by following the steps shown in the flow chart of Fig. 2. In the first step 31 the Al_2O_3 and B_4C powders are mixed. The starting mean size of the B_4C powder is from 1 to 30, preferably 5 to 15, microns. The starting mean size of the Al_2O_3 is 1 to 20 microns.

To homogenize the powders and eliminate coarse agglomerates several hundred microns in size, the powders are mixed and ground in a ball mill in the second step 33. This process permits intimate mixing of the constituents Al_2O_3 and B_4C. To aid in the comminution and homogenation, the powders are mixed in a liquid, typically deionized water. Small but effective quantities of a wetting agent, surfactant and deflocculant are added to the liquid. A small but effective quantity of a thixotropic agent may be added. The surfactant aids in imparting wetting qualities to the liquid. The deflocculant suppresses the formation of aggolmerates. The thixotropic agent imparts a fluid property to the powder when it is agitated preventing the larger particles from settling out. The powders are milled for about 1 to 2 hours producing a slurry including about 40% by weight of the powder. Organic binders and plasticizers are added to the slurry in the third step 35 and the milling is continued for ½ hour to 1 hour. The binders and plasticizers may also be added earlier in the processing, in the first or second step 31 or 33. The slurry is then spray dried in the next step 37 and screened

in step 39. The screening eliminates large agglomerates from the powder. The result of the drying and screening is to produce free-flowing spheres of 30 to 50 microns mean diameter. The spheres are predominantly Al_2O_3 with B_4C particles embedded therein. The spheres may be smaller than 30 microns or larger than 50 microns, depending on the spray-drying equipment or its operation.

Typically the slurry is spray dried in a centrifugal-separator apparatus 41 as shown in Fig. 3. Such apparatus may be procured from Niro Atomizer, Inc., Columbia, Maryland. This apparatus 41 includes a chamber 43 mounted on a movable support 45 formed of metal tubes. Under the top 47 of the chamber a rotatable centrifugal atomizer 49 is mounted. A feed device 51 for the slurry, which may be a hopper or the like, is mounted above the top 47 and is connected to the atomizer 49 through conductor 53. Heated air is supplied to dry the slurry emitted by the atomizer 49. The air is heated by a gas heater 55 and an electric heater 57 and flows to the region around the atomizer 49 through a conductor 59. Arrows 61 show the path of the heated air. The resulting mixture of gas and particulate flows through the chamber 43 and through conductor 63 as shown by the arrows 65 to a cyclone 67. The powder is separated from the gas in the cyclone and is deposited in container 69. An exhaust fan 71 controlled by a damper 73 is provided for exhausting the air as represented by the arrows 75. The air heated by the heaters 55 and 57 enters the chamber 43 at a temperature of about 300°C and is at a temperature of from 100°C to 125°C in the region of the atomizer 49.

In the next step (77, Fig. 2) the dried powder is poured into a mold 79. The mold 79 (Figs. 4-9) is of the multiple type. It includes a body 81 in which there are a plurality of cavities 83 (7 in the mold shown in Fig. 4). The mold 81 is formed of a material such as polyurethane which is capable of transmitting pressure. The mold with its cavities may itself be formed by molding

in a die. Each cavity 83 is cylindrical terminating at the top in an expanded volume 85 of circular cross section capable of accommodating a funnel 87 for depositing the powder. The diameters of the cavity 83 are precisely dimensioned. Typically the core which forms the lower portion of the cavity has a diameter which is maintained to plus or minus 0.001 inch. The diameter of this core in the lower region is typically about 0.430 inch.

A rod or mandrel 89 is precisely centered in each cavity 83. Each rod is composed of tool steel and is precisely dimensioned. The length of the rod 89 which is typically about 8 inches, for a cavity of about 7 inches, is maintained within plus or minus 0.001 inch; its diameter is maintained within plus or minus 0.0001 inch. Typical diameters of the rod 89 are 0.2870 inch and 0.2830 inch.

The funnel 87 (Figs. 6, 7) includes an outer shell 91 and an inner annular cylinder 93. The shell 91 and cylinder 93 are connected by radial plates 95. The inner diameter of the cylinder 93 typically is a slip fit on the rod 89 which extends into it. Typically the outer diameter of the cylinder 93 is 0.400 inch and is maintained to within plus 0.020 inch and minus 0.002 inch. The rod 89 is aligned in each cavity by the cylinder 93 and a precisely dimensioned groove 94 (Fig. 5) in the base of each cavity. The projection from the core which forms the groove 94 has a diameter which is maintained to within plus or minus 0.0005 and a height which is maintained to within plus or minus 0.005. Typically this diameter is 0.3100 ± 0.0005 inch and this height is 0.125 ± 0.005. The outer shell includes a tapered section 95 interposed between a cylindrical section 97 above and a thickened short cylindrical section 99, from which a cylindrical lip 101 extends, below. The lip 101 is a slip fit in the upper run of the wall 103 of the expanded volume 85 of the cavity (Fig. 8).

When the powder is to be deposited in the cavities 83, the mold is placed in a cylinder 105 (Fig. 8).

The cylinder is a slip fit on the mold 81 and serves to maintain the mold rigid and to prevent the walls of its cavities 83 from becoming deformed. The mold 81 and cylinder 105 are vibrated on a vibrating table or the like while the powder 107 (Fig. 5) is being deposited in the cavities 83 through the funnel 87. The vibration distributes the powder 107 so that it is deposited uniformly in each cavity in the annular space between the rod 89 and the cylindrical wall of the cavity. The powder is deposited along this annular space up to the junction 109 of the cylindrical portion of the cavity and the start of the taper 111 to the space of larger volume 83. The funnel 87 is removed after the powder is deposited. The rod 89 is then maintained aligned by the powder 107 and the groove 94.

After the funnel is removed a plug 113 of polyurethane or the like is inserted in the opening 85. The plug is a slip fit in the wall 103 of this opening. The plug 113 has a central well 115 into which the top of rod 89 is a slip fit.

The next step 121 is to compress the powder in the cavities 83 to form green bodies of tubular configurations. The green bodies are porous and the per cent of theoretical density is evaluated. The higher the per cent of theoretical density the less shrinkage of the tube during sintering. To produce the green bodies, cavities 83 are closed by plugs 113 and the mold 81 is placed in an isostatic press. Such a press can be procured from Autoclave Engineers, Inc. of Erie, PA. The pressure may vary from 5,000 to 60,000 pounds per square inch. A pressure of 30,000 pounds per square inch is convenient. This pressure is the limit of most typical isostatic presses.

It has been found that the increase in per cent of theoretical density of the green body with increasing pressure is not significant. This is demonstrated by the graph shown in Fig. 10. This graph was produced by compressing masses of Al_2O_3 into green bodies by applying

different isostatic pressures and measuring the per cent of theoretical density for each pressure. Per cent theoretical density is plotted vertically and pressure horizontally. The per cent theoretical density of the body compressed at about 10,000 psi was 50 as compared to 60 at about 80,000 psi. A change of 1,000 psi produces only 0.14 change in the per cent of theoretical density. The data on the Al_2O_3 applies to mixtures of Al_2O_3 and B_4C.

In the next step 123 the green body is presintered at a temperature sufficient to remove the organic binder and plasticizer. This step is optional.

The presintering step is followed by a sintering step 127. The sintering is at a temperature between 1400°C and 1800°C. During sintering the atoms of the Al_2O_3 matrix diffuse and the mass shrinks. The B_4C particles remain essentially unchanged. The sintering should be carried out in such manner that the resulting body is sintered to size requiring only a minimum of external grinding.

It is necessary to form the pellets so as to prevent excessive swelling and possible destruction of the pellets by reason of neutron bombardment and release of helium gas during reactor operation. The sintering should be carried out so that the pellets are porous. Typically the porosity should be such that the density of the pellets is equal to or less than 70% of total density. The porosity should be open so as to permit evolution of the helium. Since the density is substantially less than total density, it is necessary to control the per cent total density accurately for all boron loadings to a predetermined magnitude. It is necessary to ensure that the green bodies sinter identically, or at least predictably, from batch to batch and lot-to-lot so that the shrinkage during sintering can be controlled to obtain sintered tubes of requisite dimensions. This object is achieved by maintaining the same powder compositions and green body density during pressing and to use the same sintering schedule

including temperature, environment and time of sintering. The powders which are used must be of consistent quality.

It has been found that to achieve the desired relatively low per cent of total density ($\leq$ 70%), the sintering should take place in an inert gas such as argon at about atmospheric pressure. By sintering in argon the per cent of theoretical density can be controlled over a wide range of B_4C content. There is no appreciable vaporization. Other gases present problems. N_2 can only be used at relatively low temperatures and for short intervals, typically 1400°C, and 3 hours. At higher temperatures or for longer times boron nitride is formed. Sintering in carbon dioxide results in oxidation of B_4C to B_2O_3. Sintering in hydrogen is on the whole satisfactory but it results in lower densities of the sintered ceramic than sintering in argon. Also density of the ceramic progressively decreases with increase in temperature. Vacuum sintering can be used at lower temperatures, typically 1600°C or lower. At higher temperatures, B_4C and Al_2O_3 are lost by vaporization in the vacuum. Also, control of present or theoretical density of the vacuum-sintered ceramic is not effective.

Fig. 11 shows the relationship between the per cent theoretical density of green bodies of Al_2O_3 and B_4C and the content of B_4C in the green bodies. The Al_2O_3 was powder sold under the designated A-16 by ALCOA. The sintering was carried out in argon at 1400°C and at 1500°C for 3 hours. Per cent theoretical density is plotted vertically and weight per cent of B_4C in the green body horizontally. The per cent theoretical density rises sharply for B_4C content less than 2.5 per cent but for higher B_4C content, the change is relatively small. Between 2.5 and 25%, the per cent decreases is from 70 to 65 at 1400°C and from 71 or 72 to 64 or 63 at 1500°C. Neither the sintering temperature nor the content in the green body of above 2.5% of B_4C have a marked effect on the per cent theoretical density.

Sintering time is an important parameter where the time is substantially higher than about 3 hours. In Fig. 12 per cent theoretical density is plotted vertically as a function of sintering temperature plotted horizontally. A family of curves for three times, 1 hour, 3 hours and 8 hours are presented. The curves were plotted for ceramics composed of 80% by weight of ALCOA-A16 Al_2O_3 and 20% B_4C sintered in argon. The per cent theoretical density is substantially the same for sintering for 1 hour and 3 hours and for sintering for 8 hours at temperatures below 1500°C. But above 1500°C for 8 hours the per cent theoretical density decreases sharply with increase in temperature; i.e., the porosity rises sharply. This increase in porosity results from the reaction of B_4C with the residual oxygen in the argon-forming gaseous species at the higher temperatures during the extended heating interval. It is desirable that the argon gas be as pure as practicable for higher-temperature or extended-time sintering.

That the per cent theoretical density may be set by appropriate selection of the Al_2O_3 powder is shown in Fig. 13. In this graph per cent theoretical density is plotted vertically and temperature horizontally. A family of curves is presented. Each curve is plotted for the sintering in argon of a green body composed of 80% by weight of a selected Al_2O_3 and 20% B_4C. The green body with Reynolds 172-DBM manifests the lowest per cent theoretical density; i.e., the highest porosity; Linde-A manifests the highest per cent theoretical density. ALCOA-A16 and Reynolds HP-DBM did not differ significantly. The differences are governed by the sinterability of the powder; i.e., by the extent of the diffusion of the molecules and atoms of the powder during the sintering operation.

Changes in per cent theoretical density can also be effected by appropriate selection of the particle size of the B_4C. Higher per cent theoretical densities of the

ceramic are obtained with fine powders, typically less than 400 mesh, than with coarse powder, typically greater than 200 mesh.

In the final steps, 127 and 129 the outer surface of the tube can be ground and the pellet lengths are accurately finished to length.

The pellet lengths may also be cut from the green tube or from the presintered tube. If desired the green or presintered pellets can be machined to size prior to sintering.

Tables I and II below show typical constituents, based on 100 grams of Al_2O_3 and B_4C powder, of feed material for isostatic pressing in the practice of this invention:

TABLE I

80 grams	Linde-A Al_2O_3
20 grams	B_4C
2 grams	LOMAR PWA wetting agent
0.25 gram	CARBOWAX 200 plasticizer
1 gram	PVA (poly vinyl alcohol) binder

TABLE II

80 grams	ALCOA-A16 Al_2O_3
20 grams	B_4C
0.26 gram	TRITON-X405 wetting agent
1.59 grams	TAMOL 731 (in 25% solution deflocculant in water)
0.08 gram	SANTIZER (SANTICIZER) 160 plasticizer
0.79 gram	UCON 2000 plasticizer
2.64 grams	RHOPLEX AC33 (40% aqueous binder solution)

A slurry of the constituents in Tables I and II was formed with about 150 grams of water.

LOMAR is procured from Process Chemical Division, Norristown, NJ.

CARBOWAX 200 and UCON 2000 are procured from Union Carbide, New York, NY.

TRITON, TAMOL, and RHOPLEX are procured from Rohn & Hass, Pelham Manor, NJ.

SANTICIZER is procured from Monsanto Chemical Company

CLAIMS:

1. A method of making an annular pellet for use as a burnable neutron absorber in a nuclear reactor, characterized by mixing a first powder to form a matrix, said powder being selected from one or more of the class consisting of aluminum oxide (Al_2O_3) and zirconium oxide (ZrO_2) with a second powder of a neutron absorbing one or more of the class of elements consisting of boron, gadolinium, samarium, cadmium, europium, hafnium, dysprosium and indium or their compounds; milling said first powder and said second powder in a liquid to produce a slurry; drying said slurry to produce a dry mass of said mixed first and second powders; adding to said mass of powders means capable of adding strength to a green body to be formed of said mass of powders; depositing said mass of powders into a mold in which said mass may be subjected to isostatic pressure; subjecting said mass of powders in said mold to isostatic pressure to form a green body having the form of said pellet; and heating said green body at a temperature such as to drive off said strength-adding means and to sinter said green body to form said pellet.

2. A method according to claim 1, characterized in that the first powder is aluminum oxide and the second powder is boron carbide.

3. A method according to claim 1 or 2, characterized in that the slurry includes, water, and small but effective quantities of a wetting agent.

4. A method according to claim 3, characterized in that the slurry includes a small but effective quantity of a deflocculant in addition to the wetting agent.

5. A method according to claim 1, 2 or 3, characterized in that the strength-adding means is a small but effective quantity of a binder and plasticizer.

6. A method according to any of the preceding claims, characterized in that the first compound is aluminum oxide and the second compound is boron carbide (B_4C).

7. A method according to claim 6, characterized in that the content of the boron carbide in weight per cent in the mixture of aluminum oxide and boron carbide is from 1 to 50.

8. A method according to claim 7, characterized in that the boron carbide content is from 15 to 25 weight per cent.

9. A method according to claim 6, 7 or 8, characterized in that the initial mean size of the aluminum oxide which is mixed with boron carbide is from 1 to 20 microns and the initial mean size of the boron carbide is from 1 to 30 microns.

10. A method according to claim 9, characterized in that the initial mean size of the boron carbide is from 5 to 15 microns.

11. A method according to any of the preceding claims, characterized in that the mold is of a material capable of transmitting pressure, the said method including the step of inserting the mold in a rigid body prior to the deposit of the powder so as to maintain the mold rigid during said deposit.

12. A method according to claim 11, characterized in that the step of vibrating the mold in the rigid body during the deposit of the powder is included so that the powder is deposited uniformly.

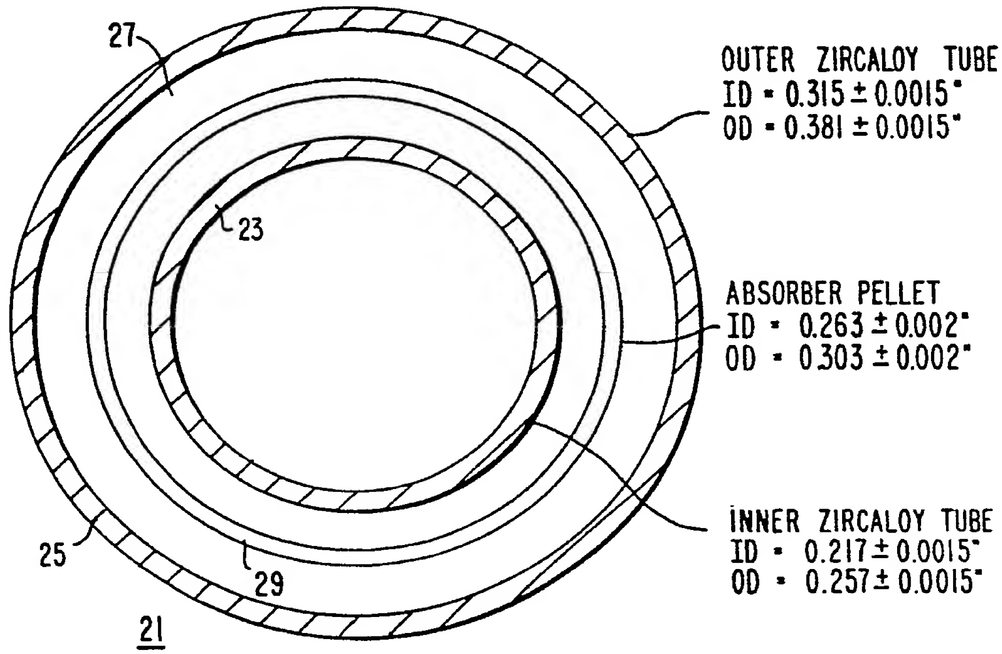

27
23
OUTER ZIRCALOY TUBE
ID = 0.315 ± 0.0015"
OD = 0.381 ± 0.0015"
ABSORBER PELLET
ID = 0.263 ± 0.002"
OD = 0.303 ± 0.002"
INNER ZIRCALOY TUBE
ID = 0.217 ± 0.0015"
OD = 0.257 ± 0.0015"
25
29
21

FIG. 1

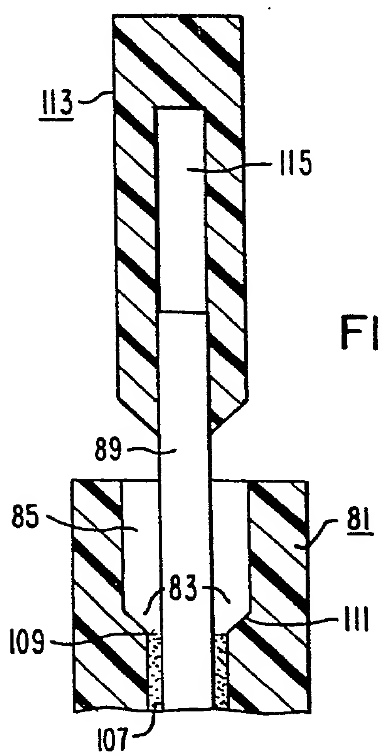

113
115
89
85
81
83
109
111
107

FIG. 9

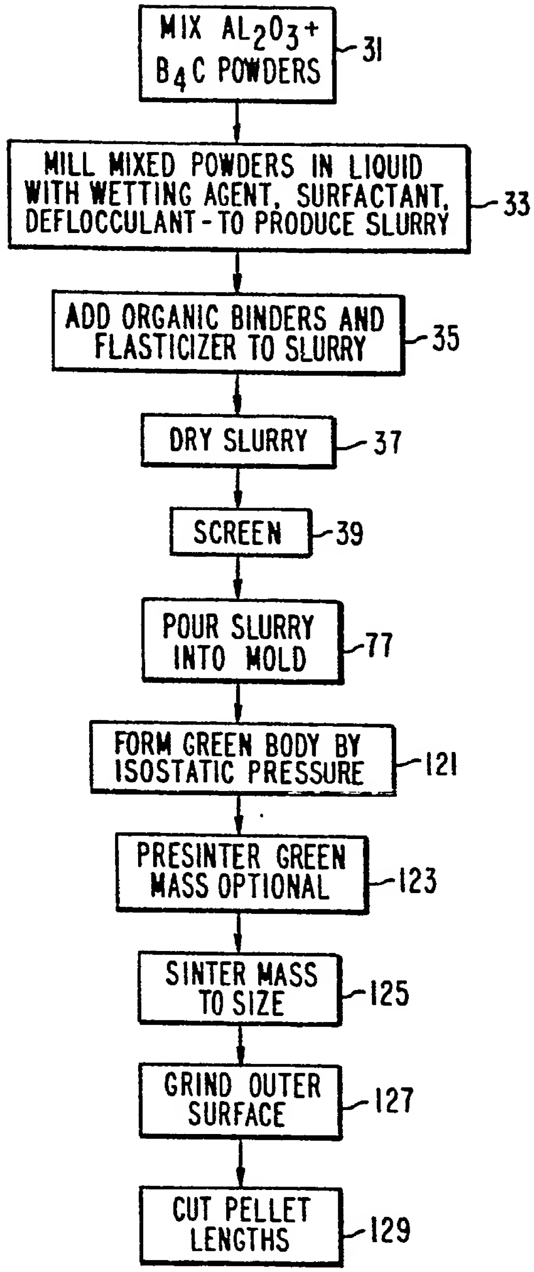

MIX AL_2O_3 + B_4C POWDERS
31
MILL MIXED POWDERS IN LIQUID WITH WETTING AGENT, SURFACTANT, DEFLOCCULANT - TO PRODUCE SLURRY
33
ADD ORGANIC BINDERS AND FLASTICIZER TO SLURRY
35
DRY SLURRY
37
SCREEN
39
POUR SLURRY INTO MOLD
77
FORM GREEN BODY BY ISOSTATIC PRESSURE
121
PRESINTER GREEN MASS OPTIONAL
123
SINTER MASS TO SIZE
125
GRIND OUTER SURFACE
127
CUT PELLET LENGTHS
129

FIG. 2

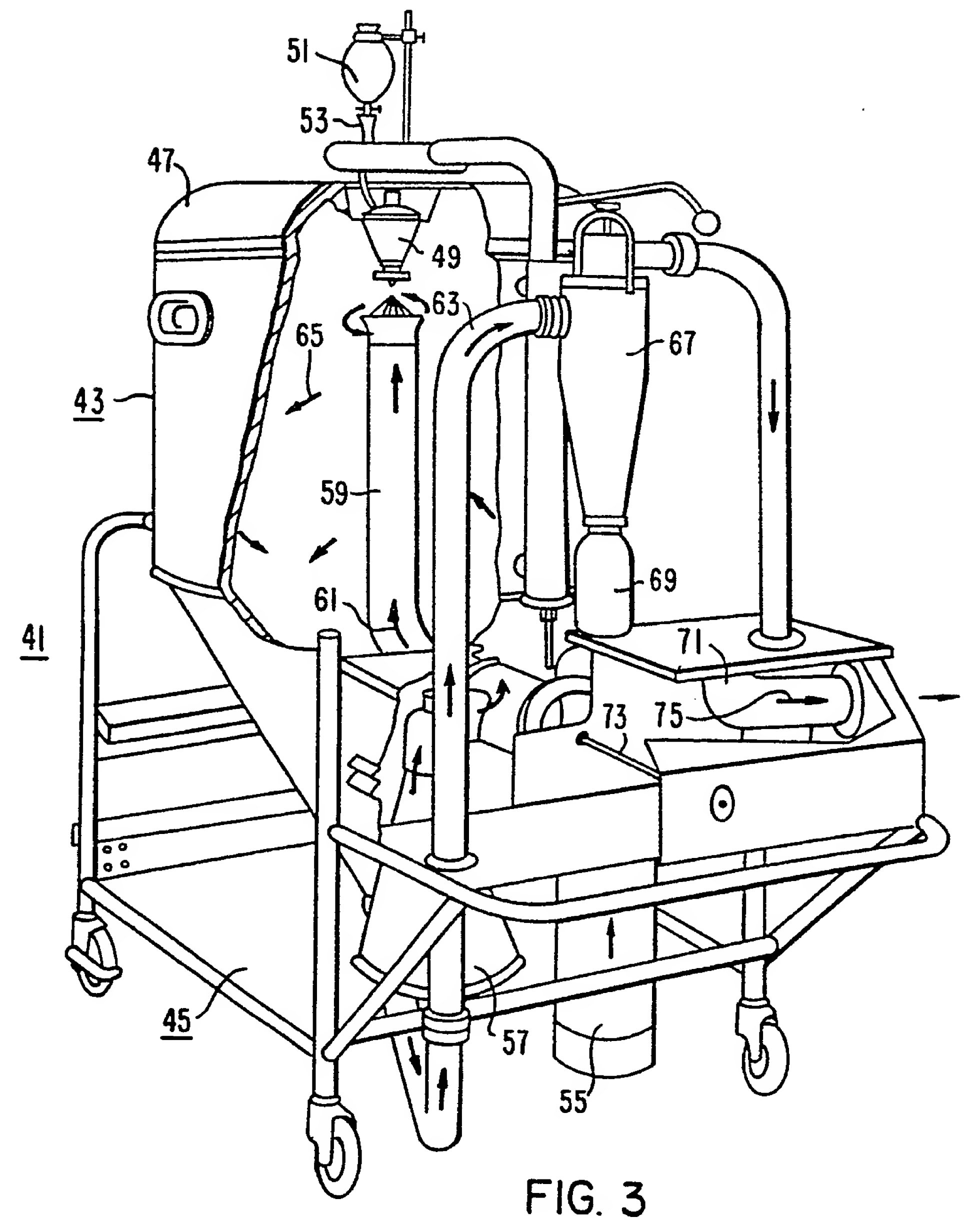


FIG. 3

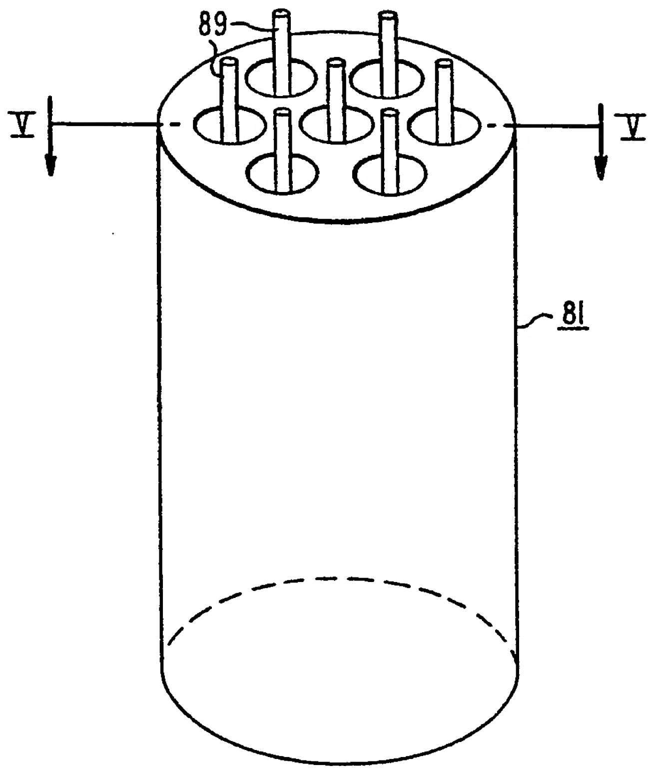


FIG. 4

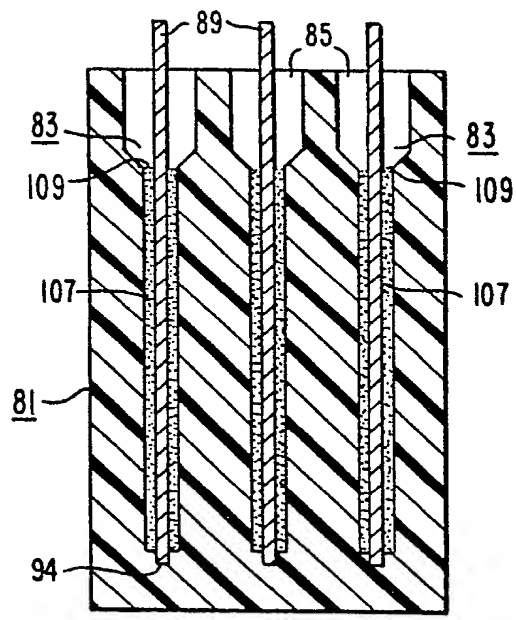


FIG. 5

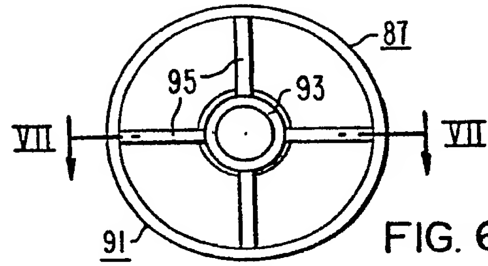


FIG. 6

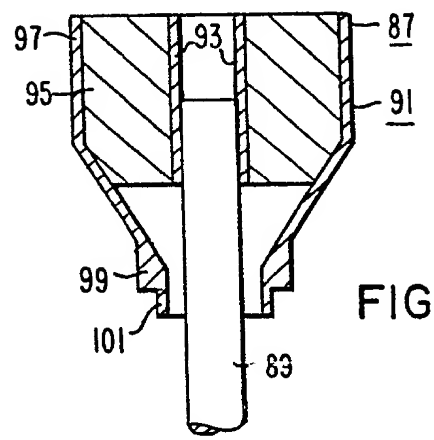


FIG. 7

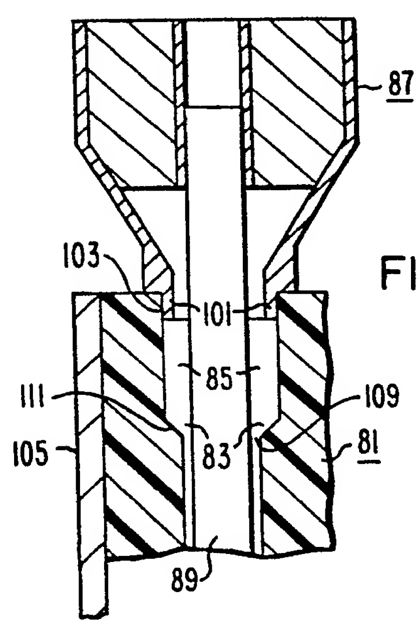
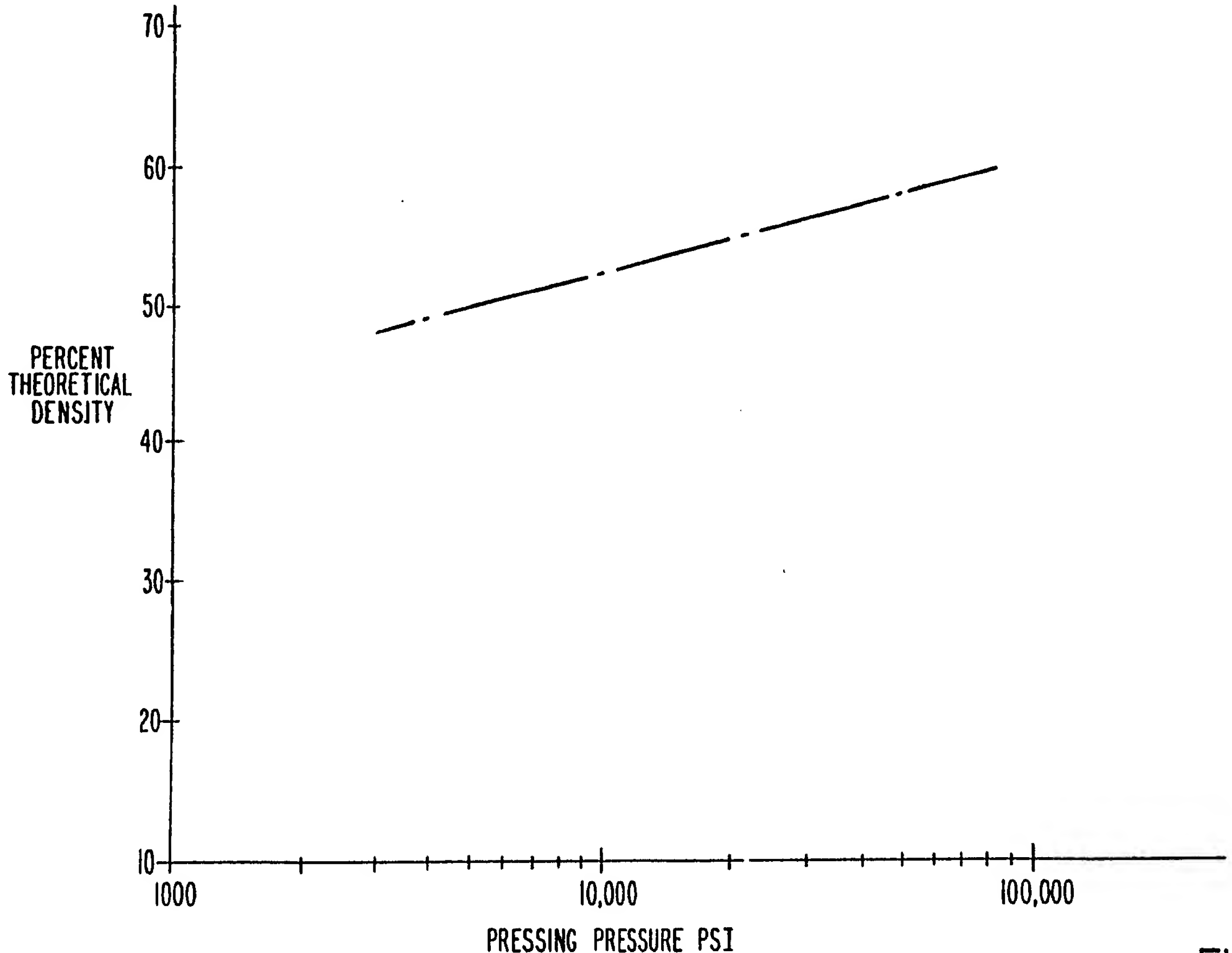


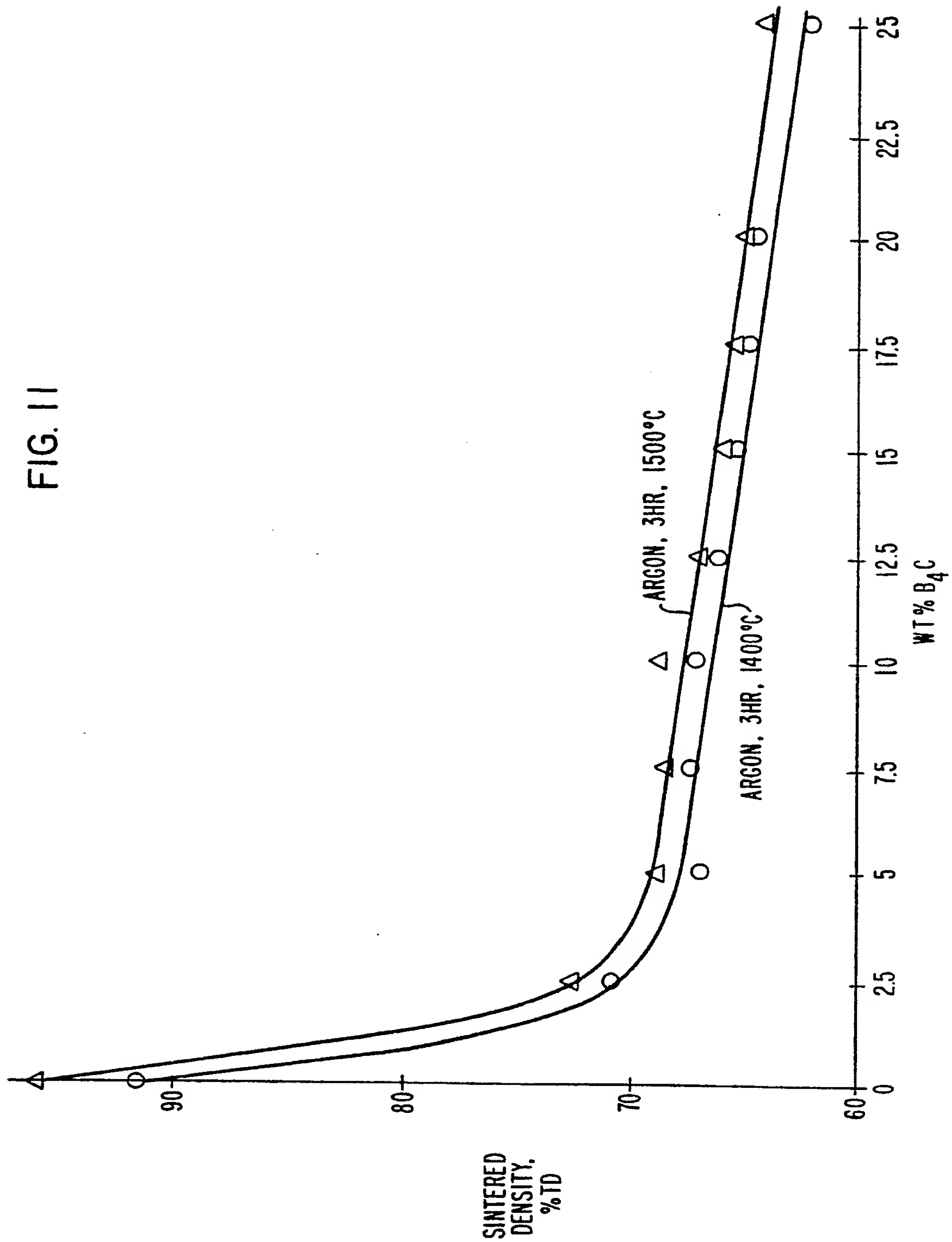
FIG. 8



PERCENT THEORETICAL DENSITY
70
60
50
40
30
20
10
1000
10,000
100,000
PRESSING PRESSURE PSI

FIG. 10

FIG. 11


SINTERED DENSITY, %TD
90
80
70
60
0
2.5
5
7.5
10
12.5
15
17.5
20
22.5
25
WT% B_4C
ARGON, 3HR, 1500°C
ARGON, 3HR, 1400°C

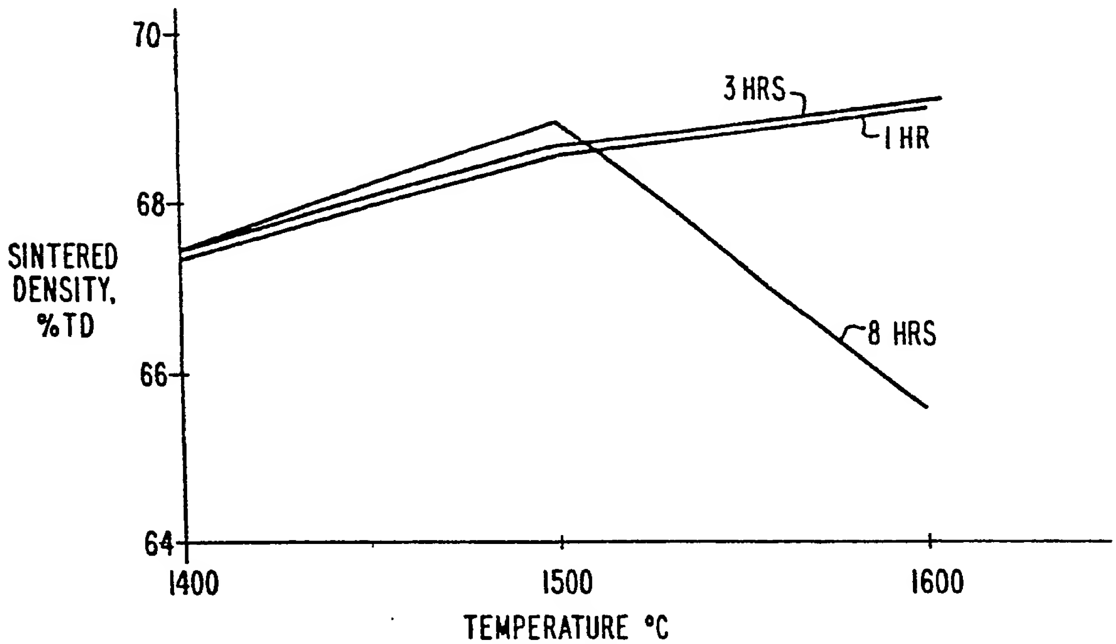

70
68
66
64
SINTERED DENSITY, %TD
3 HRS
1 HR
8 HRS
1400
1500
1600
TEMPERATURE °C

FIG. 12

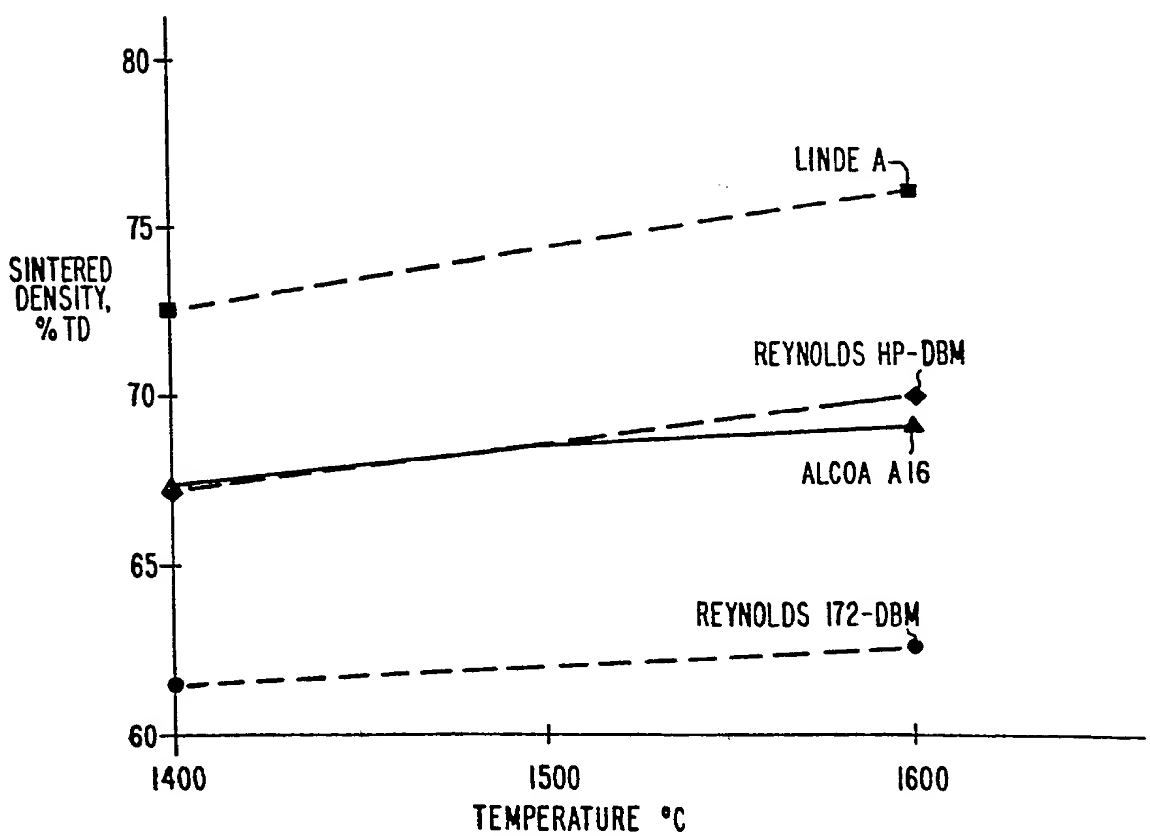

80
75
70
65
60
SINTERED DENSITY, %TD
LINDE A
REYNOLDS HP-DBM
ALCOA A16
REYNOLDS 172-DBM
1400
1500
1600
TEMPERATURE °C

FIG. 13